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Procedure for Accelerated Corrosion Testing of Aluminum Honeycomb

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SUMMARY

A testing protocol has been refined to improve the corrosion resistance testing of phosphoric acid anodized aluminum honeycomb. A comparison of different test facilities showed wide variations in the weight loss values obtained when tested per ASTM G 85 Annex A4, SO₂ salt spray test, cyclic. The ASTM G 85 Annex A4 test method was subsequently modified, requirements tightened or made more explicit, and a round-robin comparison of different test facilities showed a reduction in test variability.

INTRODUCTION

The US Navy is continually seeking ways to reduce corrosion of metal parts in its aircraft. Historically, composite sandwich structures made with aluminum honeycomb are very structurally efficient, but have been identified as being particularly susceptible to corrosion. Phosphoric acid anodized honeycomb core has been touted as having greatly improved corrosion resistance compared to conventional conversion coated aluminum honeycomb. However, standard 5% salt spray tests (such as ASTM B 117) are not sufficiently aggressive to serve as a discriminator, and the test procedure for accelerated sulfuric acid (SO₂) exposures was found to be too generic and more geared toward solid panels of metal rather than open-celled honeycomb core.

A joint program was established to identify a procedure to test for accelerated corrosion of aluminum honeycomb core that would provide comparable data. The procedure established was based on modifications to ASTM G 85 Annex A4, SO₂ salt spray test, cyclic. The participants included US Navy, NAVAIR at Patuxent River, MD; The Boeing Co, St. Louis, MO; Hexcel Composites R&T, Dublin, CA; and an independent corrosion test laboratory, Amalgatech, Phoenix, AZ.

Although the modified test procedure improved site-to-site results, inherent variability in results cannot be avoided. ASTM G 85 references ASTM B 117-97 "Operating Salt Spray (Fog) Apparatus" which states in Section 3.1, "This practice provides a controlled corrosive environment which has been utilized to produce relative corrosion resistance information for specimens of metals and coated metals exposed in a given test chamber." Section 3.3 goes on to say, "The reproducibility of results in the salt spray exposure is highly dependent on the type of specimens tested and the evaluation criteria selected, as well as the control of the operating variables. In any testing program, sufficient replicates should be included to establish the variability of the results. Variability has been observed when similar specimens are tested in different fog chambers even though the testing conditions are nominally similar and within the ranges specified in this practice."

DESCRIPTION OF PROGRAM

At the start of the program, each laboratory tested various samples of honeycomb and reported corrosion, in terms of weight loss in milligrams per square foot of exposed foil. The values varied by a factor of 7 in the same section of honeycomb (Table 1), compared to an average value for the standard product made with chromated foil.

Discussions with each test facility revealed that there was significant room for interpretation on test conditions for the test procedure in ASTM G 85, Annex A4. Agreement was reached on a coupon arrangement in the test chambers that would maximize the corrosion rate; the standard 30-day exposure would not have been acceptable because of the large number of specimens that had to be tested in a short period of time. An important part of the program was to determine exposure conditions to identify the minimum exposure time needed to discriminate amongst different types of products.

Having identified how the samples should be placed in the chamber to maximize corrosion, Boeing distributed samples of aluminum honeycomb to each participating laboratory, in a 'round-robin', to identify existing capability. The results are reported in Table 2.

These data clearly showed that simple control of sample location was not sufficient to produce similar results at different test locations. Attention was then turned to the quality of the spray, and where in the chamber atomization occurred. This was immediately followed by examining how the SO₂ gas was introduced, and how much. Each test chamber was fitted with a gas flow meter, factory calibrated for air. However, SO₂ has a density 4 times that of air, so the calibrations were not satisfactory. Consequently, each facility installed an identical gas flow meter.

Another recurring aspect of the test is that the acid will corrode other metals in the system, not just the honeycomb. This resulted in eventual failure of many parts, such as flow-controlling solenoids and atomizer tips, with consequent reduction in flow rates. Because of local variations in chamber design, no one set of written operating conditions would give corrosion values that could be duplicated in other locations; each test chamber had to be fine-tuned independently to a reference. Using Patuxent River data as reference, all test sites were able eventually to identify a flow of SO₂ to generate similar corrosion rates (Table 3).

The length of exposure was examined closely; exposure times from 5 days to 30 days were used. A consensus was reached that 30 days of exposure to acidified salt spray were substantially greater than needed, 20 days exposure were still excessive, 10 days provided good discrimination, and 5 days exposure were inadequate (Table 4).

DESCRIPTION OF TEST PROTOCOL

An interpretation of ASTM G85 implies that the test coupons could be suspended edge-on to salt spray, with atomized spray injected from one side.

However, the current experiments indicated that a more even distribution of spray occurred with a central vertical spray; the center tube is covered with an inverted conical 'hat' to create an umbrella of spray. An even rate of corrosion can be achieved on all specimens by laying them horizontally on supporting plastic rods. Figure 1 indicates the position of the spray and the location of the horizontal sample coupons. Samples from different batches of core were arranged to eliminate positional biasing, and left undisturbed throughout the test period. Every chamber run also contained a set of reference samples from the same batch of chromate-treated honeycomb.

The spray condensed into two conical flasks and the condensate tested for pH daily. Although there was a required range of pH for this effluent, there was no obvious relationship between measured pH and corrosion levels.

Clarification of test chamber set-up, coupon positioning, and movement of coupons during testing, in addition to tightening of pH requirements all helped make the test procedure more consistent. A complete description of the test procedure is attached.

Typical test procedure requires that a traveler coupon be tested in exactly the same manner, but without chamber exposure. Our experience was that any weight loss experienced by unexposed coupons was trivial, compared to that of the exposed coupons. Consequently, a traveler is not required.

CONCLUSION

Careful adherence by each test site to this common, specific test procedure, plus minimization of test chamber set-up differences can help minimize site-to-site test result differences, but there are limitations to this improvement.

SO₂ salt spray accelerated corrosion testing for aluminum honeycomb is most effective as a qualitative measure, especially if baseline aluminum honeycomb of a known corrosion resistance is exposed at the same time and in the same chamber.

RESULTS

The weight loss values of honeycomb slices, made with phosphoric acid anodized/primed 5056 0.0014in gauge aluminum foil are presented as percentages of the weight loss of blocks made with standard conversion coated foil. The nominal value for weight loss of standard product is an average of values from several different blocks, measured at Patuxent River; the lower the percentage, the better the protection.

Table 1 Initial Evaluation of Phosphoric Acid Anodized Honeycomb

Test Site	Exposure, days	Weight Loss, % of Nominal
1	10	77
2	10	11

Table 2 Round-Robin to Compare Test Sites

Test Site	Exposure, days	Weight Loss, % of Nominal
1	10	87
2	10	48
3	10	17
4	10	73

Table 3 Repeat Tests After Convergence of Procedures

Test Site*	Exposure, days	Weight Loss, % of Nominal
1	10	28
2	10	24
4	10	30

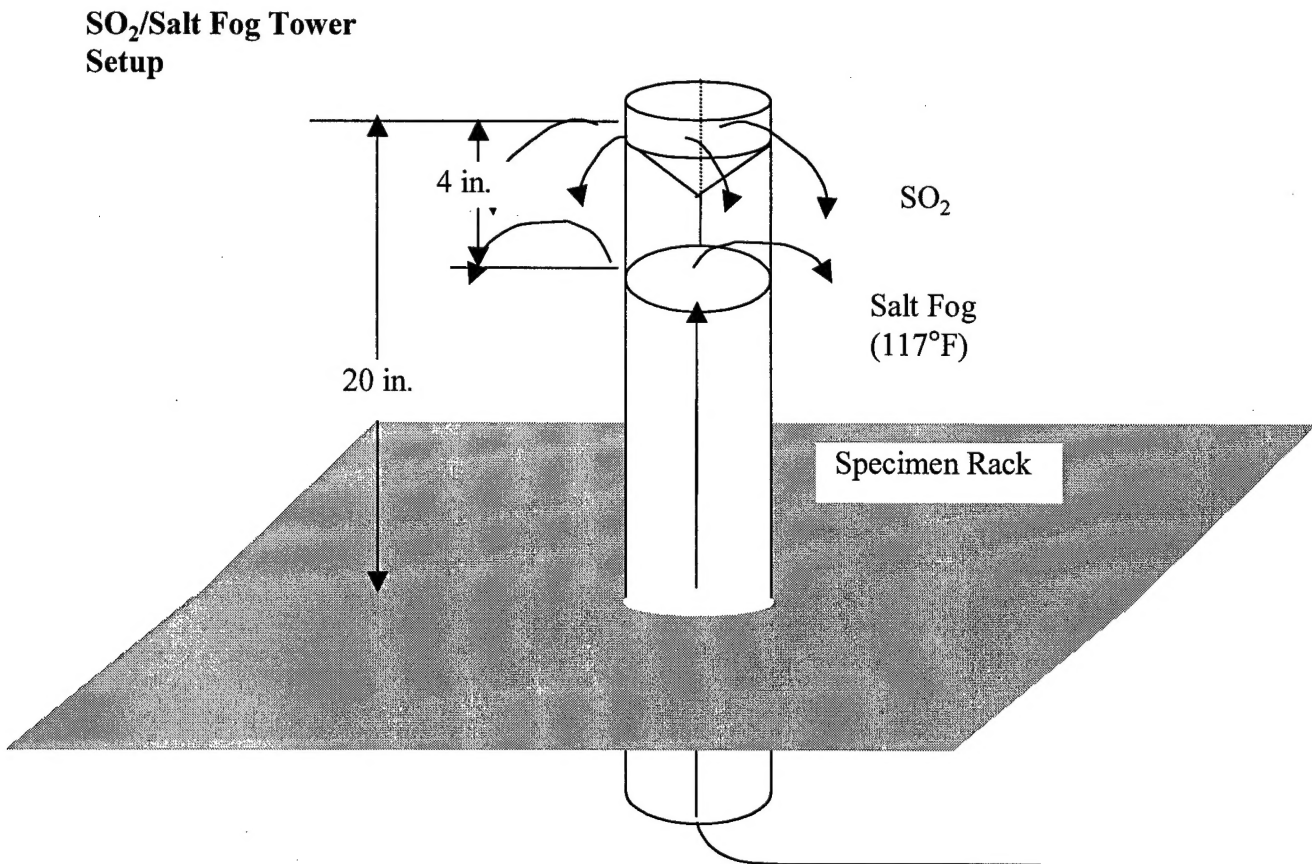
*Site 3 chamber was not adjusted for the test sample in time for this report.

Table 4 Comparison of Weight Loss with Exposure Time

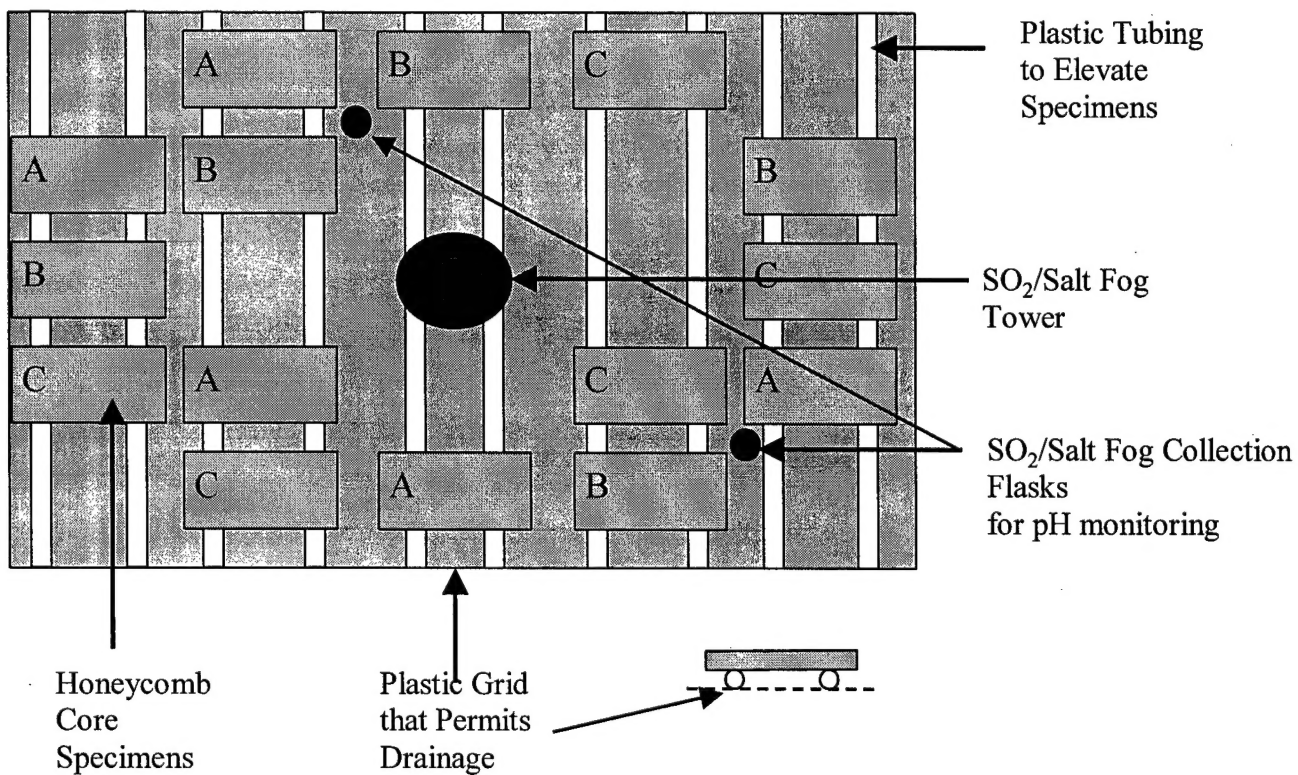
Test Site	Exposure, days	Weight Loss, % of Nominal
4	10	30
4	20	83
4	30	100*

*3 coupons survived, 2 coupons disintegrated.

Figure 1: Typical SO₂ Salt Fog Chamber Set up for Aluminum Honeycomb Core Exposure



Specimen Rack Arrangement (15 Specimens, Three Material Types)



TEST PROCEDURE

Honeycomb Core SO₂ Salt Spray Test Protocol

General

1. SO₂ Salt Spray corrosion resistance testing of Aluminum Honeycomb Core shall be conducted in accordance with ASTM G 85 - 98 and ASTM G 85 - 98 Annex 4 as modified or clarified within this document.
2. Specimen configuration, drying, cleaning, and weighing shall be conducted in accordance with Section 4.7.10 of Mil-C-7438G as modified or clarified within this document.

Specimen Preparation (ref. Mil-C-7438G, paragraph 4.7.10.1)

1. Specimens shall be $5 \pm 1/16$ inches long (longitudinal direction), $6 \pm 1/16$ inches wide (transverse direction) and 0.625 ± 0.010 inches thick. Record all three measurements for each specimen.
2. Dry specimens in an electric drying oven for 16 hours +/- 15 minutes at 350 +/- 10°F.
3. Specimens shall be removed from the oven and allowed to cool to room temperature in a desiccator.
4. Weigh specimens using an analytical balance. Record weight to the nearest milligram.

Specimen Setup

1. The sample rack location is to be located approximately 20 inches below the SO₂ nozzles (which are approximately 4" above the top of the salt fog spray tower) (See Note 2 and Figure 1).
2. Record chamber geometry and set-up, with dimensions. Use digital photos for additional clarity in the documentation.
3. Lay specimens horizontally on the sample rack in the chamber. (See Figure 1)
4. Allow for drainage under the specimens in such a manner as not to allow excessive accumulation of acidic solution at the bottom of the specimen cells. (see Note 2 and Figure 1)
5. The location of different core types in the chamber shall be randomized in such a way that chamber location will not favor one type of core. (see Note 2 and Figure 1)
6. Record position and orientation (degrees from vertical, etc.) of each specimen. Use digital photos for additional clarity in the documentation.
7. Record date and time that flow is turned on in the chamber (exposure begins).
8. Do not disturb specimens once in chamber until exposure is complete.
9. Do not shake off or remove SO₂ salt spray condensate from specimens until exposure is complete.
10. Record date and time that flow is turned off in the chamber (exposure is complete).

Chamber Conditions

1. Introduction of the salt spray and SO₂ gas shall be from a central dispersion tower to ensure uniform distribution of the spray and gas. Use of a dual tower chamber is acceptable.
2. The baffle or nozzles should not impinge directly on any of the test specimens.
3. Chamber conditions shall consist of continuous salt spray with introduction of the SO₂ gas for 1 hour, 4 times daily (every 6 hours) or some other suitable cycle to achieve the required accumulation rate and pH level, as described below in item #9.
4. The salt-water formulation shall be a sodium chloride solution in accordance with ASTM G 85, paragraph 6.3. Ensure that the pH of the salt solution is measured per ASTM G 85, paragraph 6.4 and Note 6. Record the pH of the salt solution.
5. The saturation tower (bubble tower) shall be 117 +/- 2°F, per ASTM G 85, paragraph A4.3.1. Record tower temperature in accordance with ASTM G 85, paragraph 4.3.1 and Note 2.
6. The exposure chamber temperature shall be 95 +/- 3°F, per ASTM G 85, paragraph A4.4.1. Record chamber temperature in accordance with ASTM G 85, paragraph 4.3.1 and Note 2.
7. The equipment and materials required for the addition of the SO₂ gas into the cabinet is specified in paragraph A4.4.2 of ASTM G 85. Record the purity of the SO₂ gas used.
8. The exposure chamber may be opened periodically during the test for the purposes of inspecting and cleaning the SO₂ delivery holes as described in #9 below, and for acidity and flow rate accumulation measurements as stated below in item #10. However, the specimens shall not be moved or disturbed during these processes.
9. Once every two or three days, the exposure chamber shall be opened and the "spray ring" through which SO₂ gas is introduced into the chamber shall be inspected for clear and unobstructed flow. Time between inspections shall not exceed 72 hours. If any of the delivery holes have become clogged with salt crystals or other material, all of the delivery holes shall be reamed clear. Record date and time of inspection, and results of inspection.
10. Fog shall be collected per ASTM G 85, paragraph 4.3.2 and Note 3 (see also Figure 1 below). Remove the flasks from the chamber and measure both the accumulation rate and the acidity of the condensate. Measure the pH using a pH probe per ASTM E 70. The period of time between flask removals shall be at least 16 hours but not more than 72 hours. Record both the pH measurement and the accumulation rate for each flask, along with the date and time. The pH of the collected solution shall be 2.8 ± 0.2 ; the accumulation rate shall be between 1.0 and 2.0 ml of solution per hour per 80 cm² of horizontal collecting area.

Post Test Cleaning and Weighing

1. Rinse specimens gently and thoroughly for at least 5 minutes in clear running water no greater than 15 minutes after removal from the SO₂ salt fog chamber.
2. No greater than 15 minutes after rinsing, the specimens shall be stripped in a phosphoric-chromic acid solution for $5 \pm 1/4$ minutes at $212 \pm 2^\circ\text{F}$.

The solution shall consist of the following: (ref. Mil-C-7438G, paragraph 4.7.10.2)

Phosphoric acid, 85%	103 ml
Chromic acid (CrO ₃)	76g
Water, to make	3.75 liters

3. The specimens shall be removed from the solution and rinsed in distilled or deionized water for a minimum of 5 minutes.
4. If corrosion products are still evident on any of the specimens, repeat steps 2 through 3 for these specimens one time. If corrosion products remain after second stripping, record observation and continue with step 5.
5. Dry specimens at $225 \pm 5^{\circ}\text{F}$ for 35 ± 5 minutes.
6. Allow specimens to cool to room temperature in a desiccator.
7. Weigh specimens using an analytical balance. Record weight to the nearest milligram.

Weight Loss Measurement (ref. Mil-C-7438G, paragraph 4.7.10.3)

1. Calculate the weight loss in mg/sq. ft. of exposed foil area using the following equation:

$$M = (36C(O-A))/(TLW)$$

M = Weight loss in mg/sq. ft. of exposed foil area
C = Nominal cell size (inch)
T = Thickness measurement in direction of cell axis (inch)
L = Ribbon length direction (inches)
W = Transverse direction (inches)
O = Original weight of specimen prior to exposure (mg)
A = Final weight of specimen after stripping (mg)

Notes

1. The flow meter and corresponding capability used by NAWCADPAX is as follows:

Flow meter:

Manufacturer: Linde Corporation (Division of Union Carbide) tube number 201-4331
(glass)

Local Rep: Keystone Airgas (800) 692-7237 part number - FM 43111

Flow range: 0.003 to 0.062 liters per minute

Flow Setting: 35-50 (0.011- 0.017 liters/min)

2. Figure 1 is the recommended specimen layout for 15 specimens consisting of 5 each of 3 different core types (A, B, and C). If all three different core types are not being tested, simply leave those slots empty.